

Voltage and Current Transients in Lithium Thionyl Chloride Cells Accompanying Interrupted Electrolyte Additions

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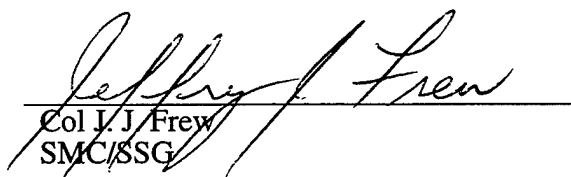
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13. ABSTRACT (Maximum 200 words) An investigation was carried out in an attempt to explain the out-of-family voltage trace associated with one of the 250 A-h lithium thionyl chloride cells used in batteries for selected Centaur missions. The voltage trace was generated during the manufacturing step where electrolyte is added to the cell. The investigation was carried out in several steps. First, discussions were held with the manufacturer, who was able to suggest theories that were consistent with the observations that were available for review. These theories were later tested in the electrochemical laboratories of The Aerospace Corporation. Using specially constructed cells equipped with reference electrodes, we measured the individual contributions of the anode, cathode, and cell container to the overall voltage transients associated with an interrupted cell filling process. These results validated some of the manufacturer's theories. What had been suggested as a minor contributor to the voltage traces was found by experimentation to be the most important one. That was the contribution of the porous carbon electrode. Our experiments added further support to the earlier conclusion that the out-of-family voltage signature seen in cell #474 was not indicative of a problem within the cell.				
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1. Introduction

During the process of filling large lithium thionyl chloride cells, the voltage between each of the two anode terminals and the single insulated cathode feedthrough are monitored and recorded on a strip chart. During the review of the strip chart recordings of all the cells that went into a completed nine-cell battery, it was noted that the signature for one of the cells was out-of-family. The cell manufacturer did not have an immediate explanation for this signature. Upon further researching of their records and the memories of their staff members involved with the filling of the cell in question, it was discovered that the filling process had been interrupted due to a blockage problem. The blockage occurred in a narrow tube through which the electrolyte solution is drawn into the cell as it is filled. This resulted in an interruption in the flow of the electrolyte into the cell. Following the time delay as prescribed by the filling procedures, the problem was eliminated, and the flow of fluid into the cell was resumed. It was at this time that the out-of-family voltage signature began. Figure 1 illustrates what is considered to be a nominal filling signature and the out-of-family signature. The concern on this issue is related to the possibility of a momentary short circuit being responsible for the drop in voltage during the filling of a lithium thionyl chloride cell. A fully developed short circuit in this type of cell results in the bursting of the pressure release disc or sometimes an explosion. Evidence in support of an intermittent short circuit or a short that burned itself clear would indicate a cell that,

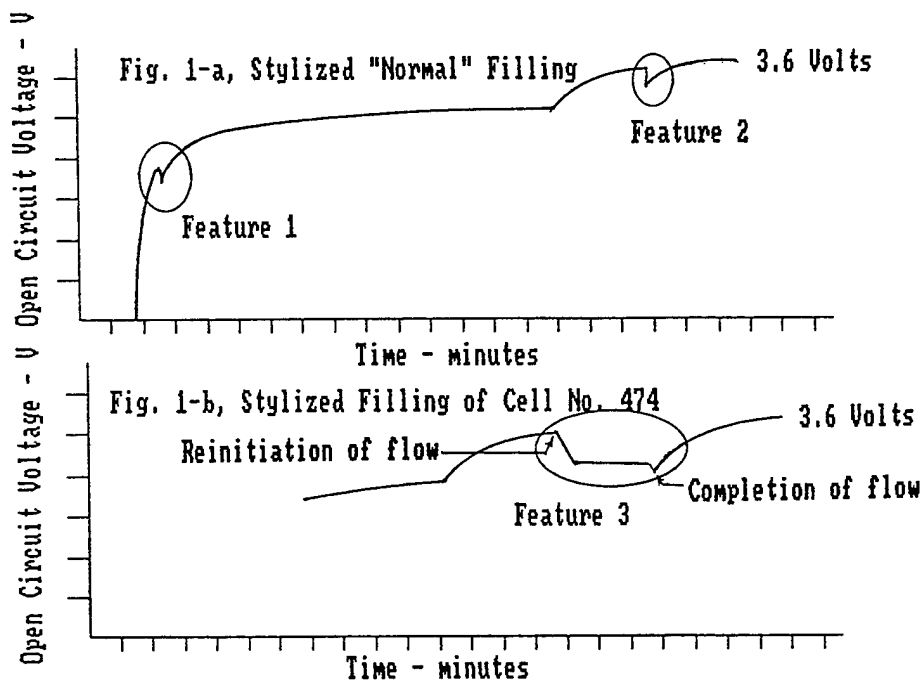


Figure 1. Stylized open-circuit voltage traces during electrolyte filling process.

although free of a short circuit, might be prone to reestablishing a destructive short if the battery would be subjected to the environmental rigors of launch. As the manufacturer reviewed the historical records of filling cells of this general type, several other instances of interrupted fillings were found. However, none of these exactly matched the signature of the cell in question.

For these reasons, further dialogue related to the chemical and electrochemical aspects of a lithium thionyl chloride cell during the filling process was held with the manufacturer. This was done by reviewing the possible transient voltage and current responses one might expect from several different hypothetical laboratory-type cell configurations. These cells would be equipped with reference electrodes and would contain different combinations of electrodes. Following this interchange covering four or five different cell arrangements, a tentative understanding of the functioning of the cell components as they are exposed to the thionyl chloride electrolyte was reached.

It was concluded that the stainless-steel cell container functions as a rapidly pacifying cathode that is connected to the lithium electrode. The lithium electrode is functioning either as a normal lithium electrode or as a combination lithium anode and a rapidly corroding cathode in its freshly exposed areas. A mixed potential is established during the transient time. During the reinitiation of the filling procedure, the lithium electrode responds as an electrode with a mixed potential between a lithium electrode that has already established an operating passivation layer and a rapidly corroding thionyl chloride redox electrode on the freshly exposed portions of the lithium surface. The newly exposed portion of the stainless-steel cell case again acts as a rapidly pacifying cathode connected to the lithium electrode. The manufacturer stated that the porous carbon electrodes are not subject to any time-dependent responses during the filling or interrupted filling processes. Due to its partially nonwetting nature, it is not known what the degree of saturation or rate of wetting would be during the first few minutes over which the filling signatures are gathered.

It was felt to be instructive to construct some of the cell configurations that were discussed with the manufacturer to see how accurate the predictions concerning their transient voltage and current behaviors would be. Figure 2 shows the general arrangement of electrodes within the type of cell that had experienced an interruption during its filling procedure. A series of anodes, cathodes, and separators are stacked horizontally within the cell. The electrodes are attached to bus bars located near the outer periphery of the cell. The dielectric feedthrough for the cathode bus bar isolates the cathodes from the remainder of the cell components. The negative terminal is not a dielectric material, and thus the cell container is held at the potential of the lithium electrodes. Figure 3 shows these features in a more simplistic manner. The lithium is depicted as being protected by a layer of lithium chloride, and the electrically isolated carbon cathode is shown as a highly porous structure offering a large surface area where the thionyl chloride can be reduced during cell discharge. As a first approximation, the electrochemical behavior of the three components can be understood in the following paragraphs.

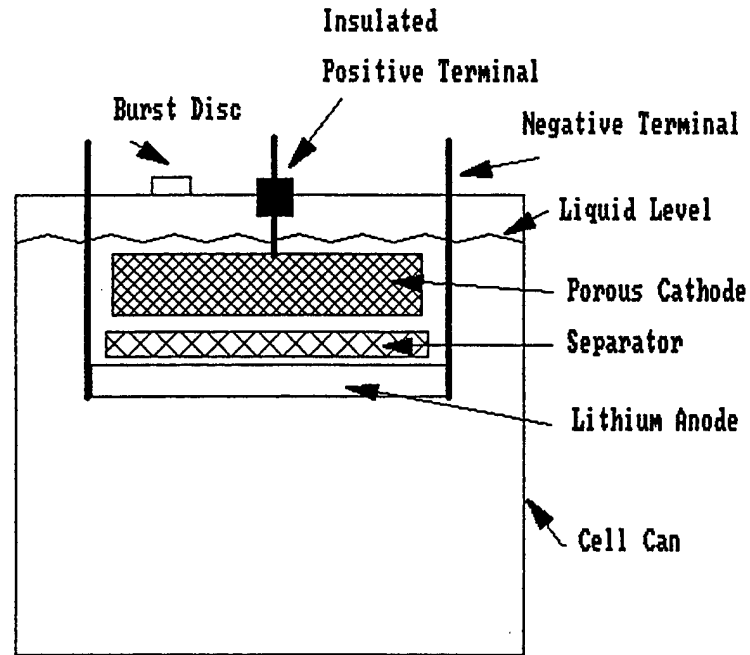


Figure 2. Schematic of a large lithium thionyl chloride cell.

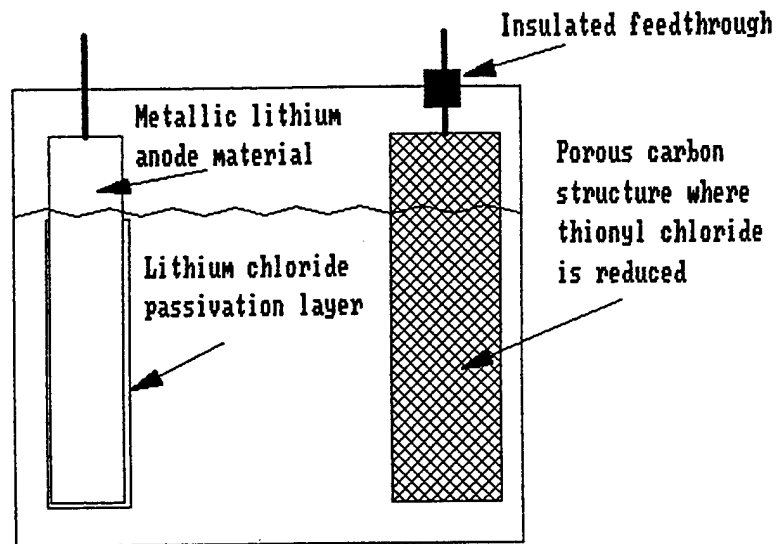
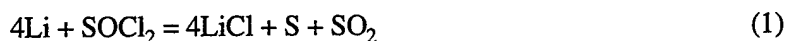


Figure 3. Schematic of a lithium chloride cell in the "can-negative" configuration.

2. Electrochemical and Chemical Behavior

2.1 Lithium Electrode

When a fresh lithium surface is exposed to the oxidizing power of thionyl chloride, the surface is rapidly corroded as it forms a protective layer of lithium chloride. The reaction is described in Eq. (1).



Although the extent of this reaction is essentially complete in a matter of seconds, it continues at a progressively lower rate for several days or even weeks. This corrosion layer is slightly conductive to lithium ions. At low current densities, the lithium ions formed at the interface between the metallic lithium and the protective film of lithium chloride can move through the film and into the electrolyte. At higher current densities, portions of the layer are removed, and the process of reforming the protective layer begins again.

2.2 Stainless-Steel Cell Container

The stainless-steel portions of a cell are also protected by a passivation layer of lithium chloride as a result of being oxidized by thionyl chloride. If the cell container is not connected to the cathode or the anode (this is called case neutral), a chemical reaction between the stainless-steel and the thionyl chloride takes place, forming a passivation layer protecting the metal. If the container is in contact with the lithium anode (referred to as case negative), the surface functions as a low-surface-area cathode at which small amounts of thionyl chloride are reduced. Since it is connected to the anode, a complete electrochemical cell is formed, and discharge of the active materials begins. As a passivation layer forms to protect the container material, the rate of the electrochemical reaction is reduced to an almost negligible rate.

2.3 Porous Carbon Cathode

The porous carbon material used as the cathode was felt to be a potential source of the voltage transient under investigation. As a result of the manufacturing processes for carbon blacks, the surface of the carbon might contain many active surface sites that could be oxidized by the thionyl chloride. Since most of these sites are located within the porous structure of the cathode, it would take a period of time before the reaction products could diffuse away and equilibrate with the bulk electrolyte.

The half-cell potential present at the cathode can be described by a standard Nernst relationship.

$$E = E^0 - (nF/RT)\ln[\text{SOCl}_2]^2/[\text{S}][\text{SO}_2][\text{Cl}^-]^4 \quad (2)$$

When measured against a passivated lithium electrode, the thionyl chloride voltage is about 3.65 V. When a mixture of all four components is present, the voltage will be a function of the concentrations of these four components as dictated by Eq. (2).

3. Experimental Studies

Following the discussions with contractor personnel concerning the expected responses of several different cells equipped with reference electrodes, the possibility existed that the transient filling characteristics of these cells were due, in part, from the lithium electrode, the carbon electrode, and the stainless-steel cell container. In a completed cell containing all three components and without a reference electrode, it was impossible to partition the effects of any one component. The experimental plan was to construct cells with different combinations of electrodes (including a reference electrode) in order to differentiate the transient behavior of each of these three possible contributors. Further, several arrangements were specially configured to measure the current flow that would be present during the transient voltage responses of the cell components.

3.1 Materials and Equipment

1. **Lithium**—Battery grade lithium foil material as supplied by Lithcoa.
2. **Electrolyte**—A standard 1.5 molar solution of lithium tetrachloroaluminate dissolved in thionyl chloride as supplied by Lithcoa.
3. **Cell Container**—Material representative of the cell container were strips of stainless-steel alloy 304L.
4. **Cathode**—The porous carbon cathode material was a mixture of carbon black and Teflon emulsion that had been pressed onto a stainless-steel screen and heated to sinter the Teflon. The cathodes used in these studies were not the same as those used in the cell in question, but were felt to be generically similar.
5. **Experimental Setup**—Experiments were carried out in a glove bag filled and purged with argon.
6. **Data Collection**—Current and voltage readings were recorded every 100 ms and stored on a computer disc for later retrieval and plotting.

The different elements that were arranged several different ways are depicted schematically in Figure 4. The reference electrode was a strip of lithium that had been equilibrated for at least five minutes with the electrolyte. This resulted in an adequate protective layer and permitted the voltage of this electrode to be free from any time-dependent transients. Since the objective of these tests was to explore the transient response characteristics that might accompany an interrupted fill process, approximately 10% of the electrolyte was initially added to each cell. Following a waiting period of one to two minutes, the remainder of the electrolyte was added. The following paragraphs and accompanying figures explain the different cell configurations and the objective for each test.

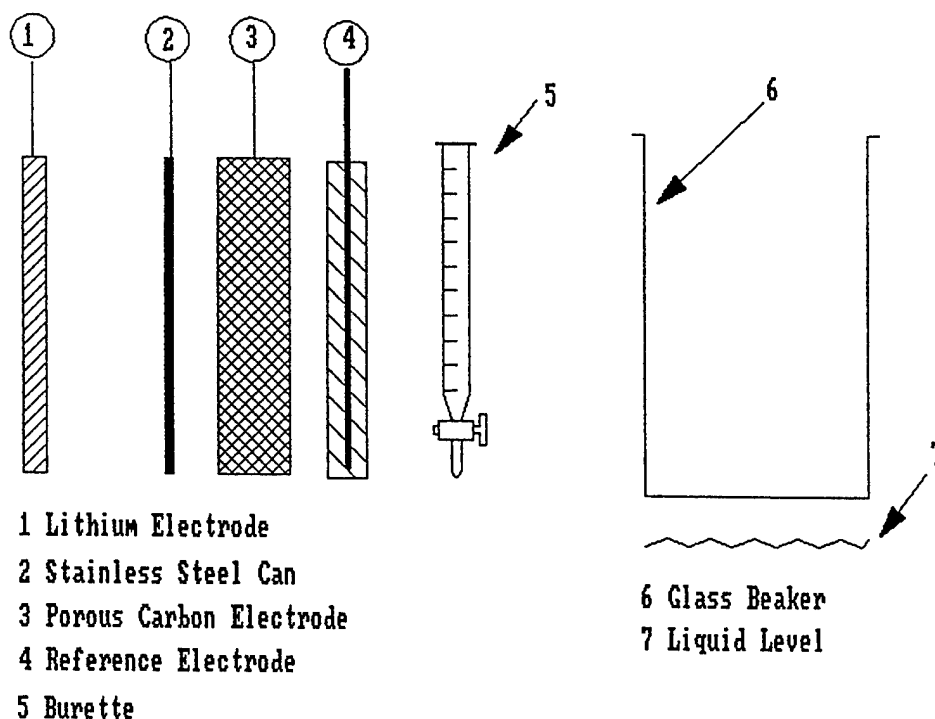


Figure 4. Schematic of cells parts used to conduct experimental cell studies.

3.2 Cell Configurations

3.2.1 Mixed potential associated with the lithium electrode

The cell shown in Figure 5 permitted the measurement of the transient voltage present at the lithium electrode during the initial fill of approximately 10% of the electrolyte, followed by the response during the filling of the remainder of the cell. When the second portion of electrolyte is added, the upper portion of the lithium will function as a redox cathode for a short period of time until it becomes passivated.

3.2.2 Cell container is electrically shorted to the lithium

The cell in Figure 6 is used to examine the situation where a portion of the lithium electrode is first allowed to develop a passivation layer. When the remainder of the electrolyte is added, the stainless-steel strip can function as a redox cathode connected to the lithium. As the stainless steel develops its own passivation layer, the current should diminish and the potential between the lithium and the stainless steel should approach zero.

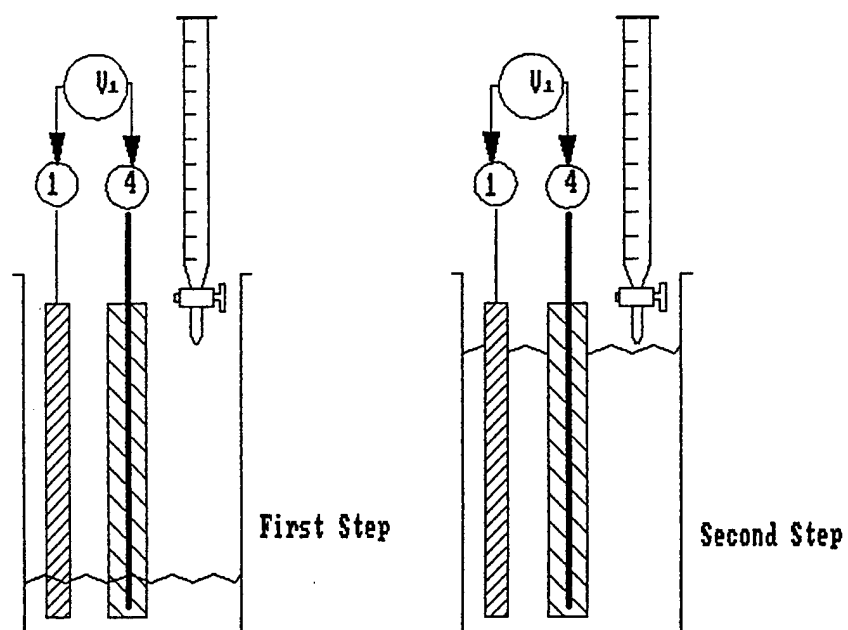


Figure 5. Cell used to examine the mixed potential transient at the lithium electrode.

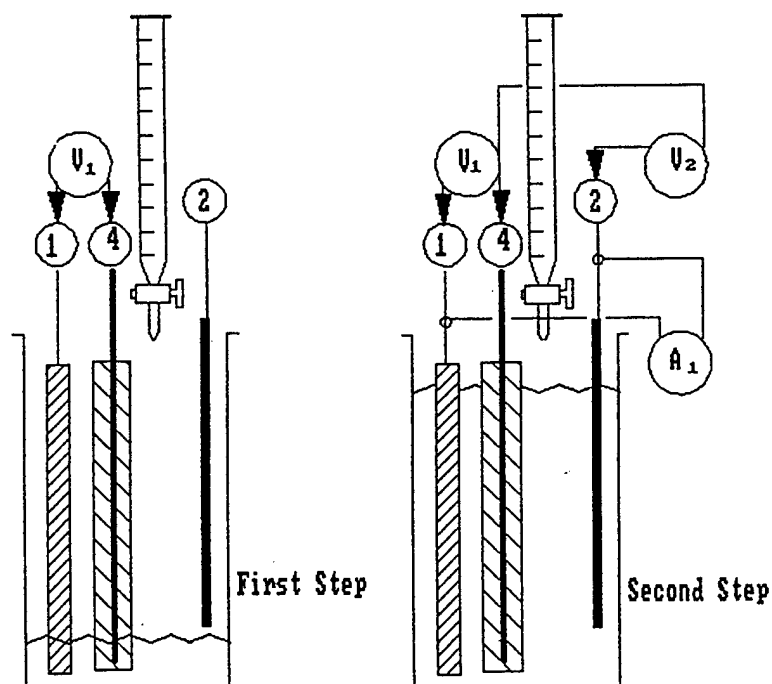


Figure 6. Cell used to measure the voltage and current transients caused by stainless steel being connected to the lithium anode.

3.2.3 Belated touching of can by the electrolyte

As shown in Figure 7, a complete cell is first established at the lower portions of the lithium and carbon electrodes. Once established, the remainder of the electrolyte is added. As the stainless steel is exposed to electrolyte, its surface is able to support the electrochemical reduction of thionyl chloride as the lithium to which it is connected is oxidized. The transient current can also be measured.

3.2.4 Search for cathode transient

In Figure 8, the electrodes are configured so the transient voltage phenomena at or within the porous carbon cathode can be determined. Positioning a reference electrode between the anode and cathode permits the relative contributions of the two electrodes to be ascertained.

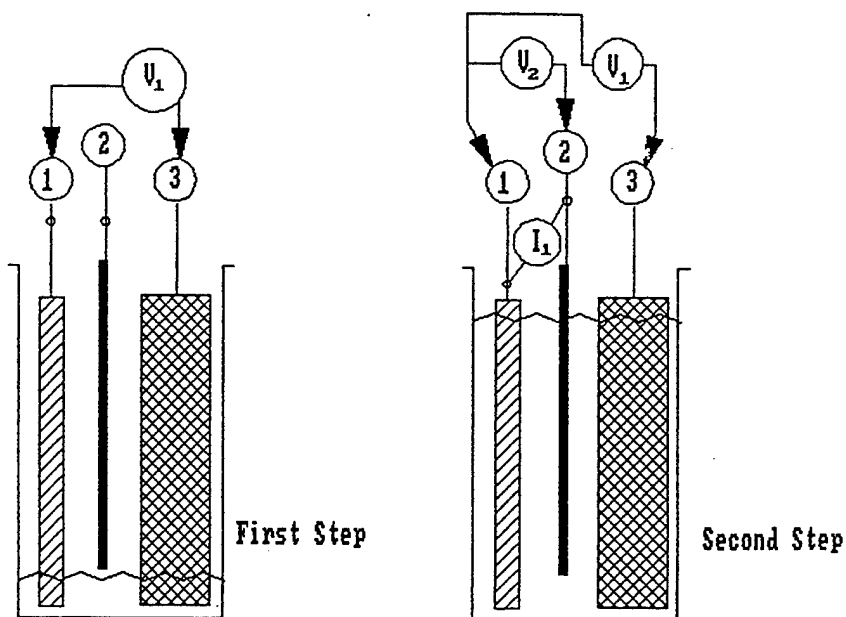


Figure 7. Cell used to simulate the stainless steel belatedly coming in contact with the electrolyte.

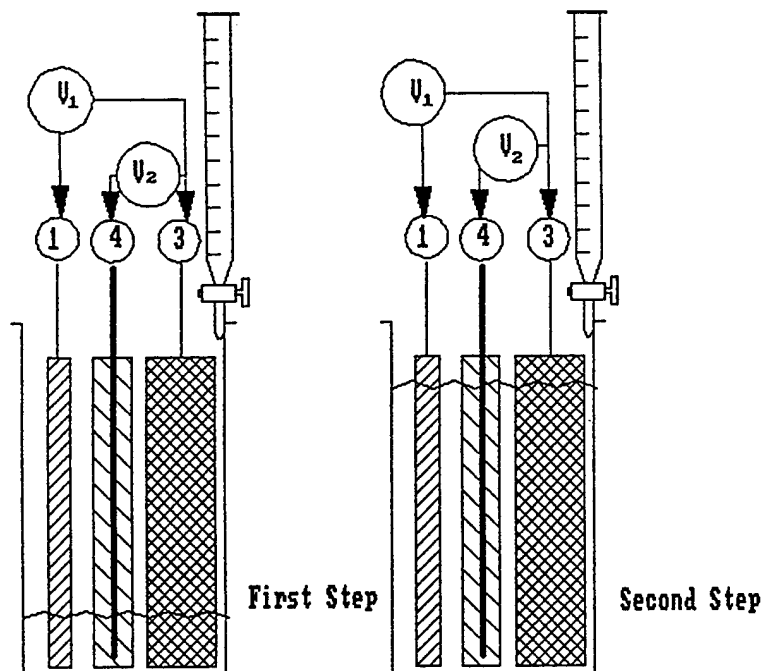
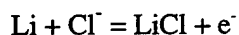


Figure 8. Cell used to examine the voltage transients associated with the porous carbon electrode.

4. Results

4.1 Mixed Potential Associated with Lithium Electrode

Figure 9 shows the voltage transient occurring at the lithium electrode as the second segment of the electrolyte is added to the cell. It shows a very sharp rise, going cathodic by about 135 mV and returning very close to the reference electrode within 4 to 5 s. This behavior is explainable with the help of Figure 10. This figure depicts the chemical and electrochemical reactions taking place during the transient time. Once a stable passivation layer is formed in areas that have been exposed to the thionyl chloride electrolyte, and new areas are subsequently exposed, the newly exposed areas function as an electrode for the electrochemical discharge of thionyl chloride while the previously exposed areas are electrochemically discharged. Electrons from the previously passivated portion of the lithium are supplied to the portion that is discharging thionyl chloride. As the passivation process progresses, the amount of current flowing between the two portions of the lithium diminishes. The reaction taking place at the lower portion of the electrode is described in Eq. (3). An electrode surface that functions only to interchange electrons to soluble species within the electrolyte is generally referred to as a redox electrode.



Eq. (3)

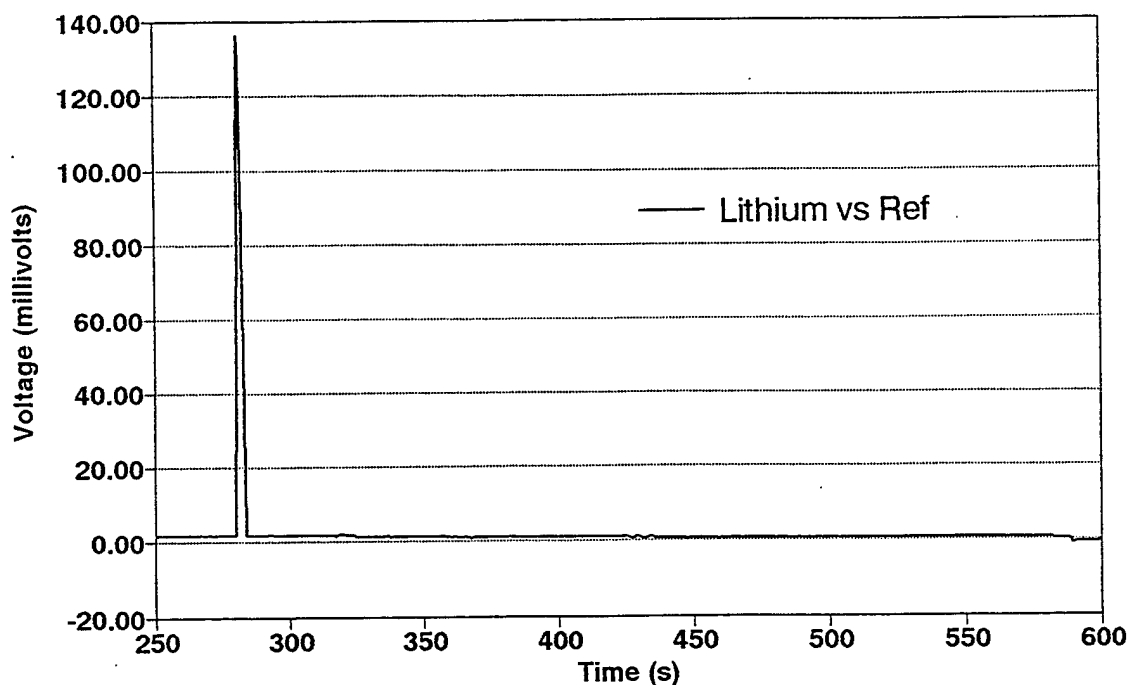


Figure 9. Transient mixed potential at the lithium electrode recorded during the second addition of electrolyte to the cell in Fig. 5.

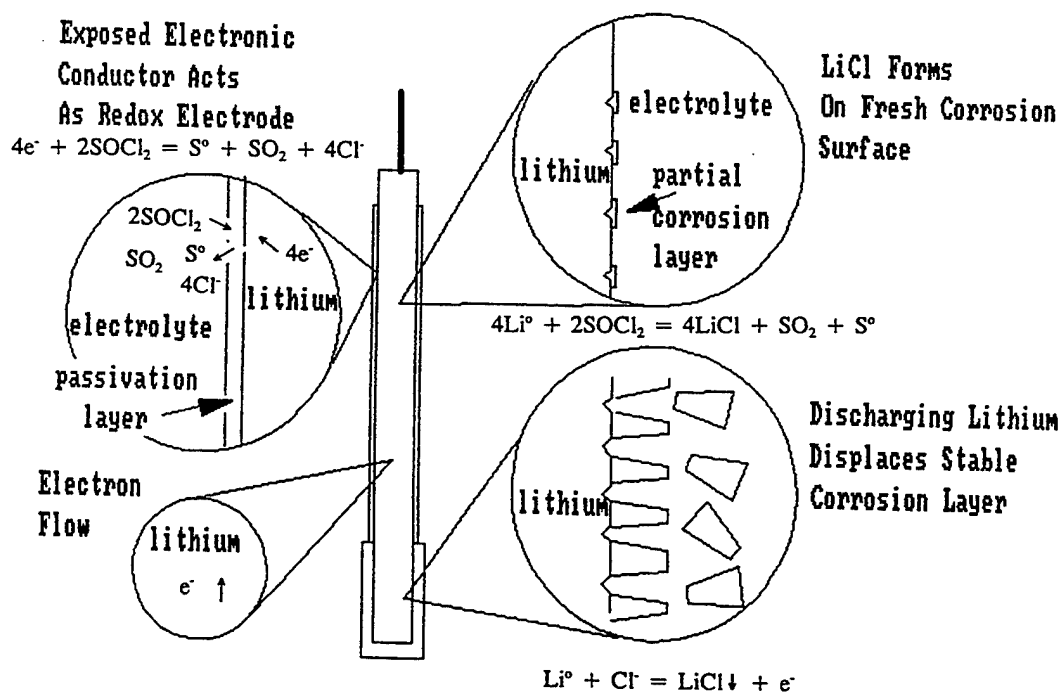


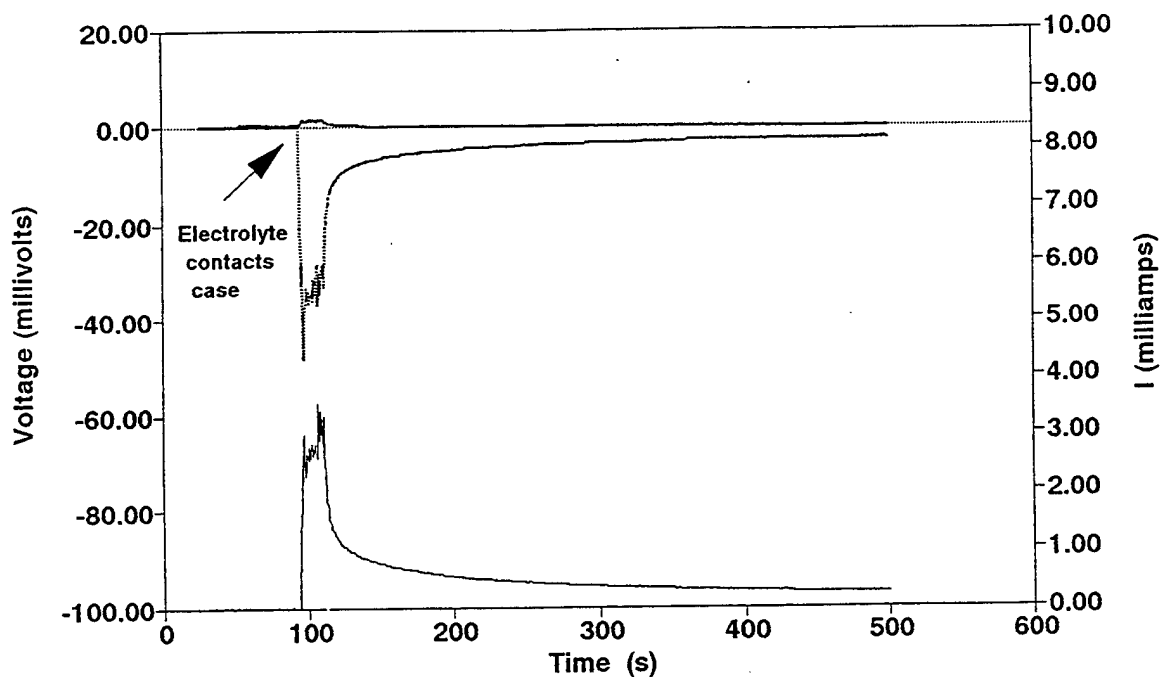
Figure 10. Schematic of suggested chemical and electrochemical reactions occurring at the upper and lower surfaces of a lithium electrode following the addition of the second increment of electrolyte.

The reaction taking place at the newly exposed surface is actually made up of one electrochemical reaction, Eq. (4), and the corrosion reaction described in Eq. (1).



4.2 Cell Container is Electrically Shorted to the Lithium

Figure 11 shows the current between the lithium and the stainless steel. The potential of the lithium is reduced due to the current flow. The current approaches zero after an extended period of time. Figure 12c depicts the stainless steel functioning as a rapidly pacifying redox electrode for the reduction of thionyl chloride. For the situation where the cell container is not connected to the anode, then the "can-neutral" case, Figure 12b, would be the accurate representation of the passivation process.



— V (Li vs Ref) — V (Li vs case) — I (Li to case)

Figure 11. Voltage and current traces simulating the shorting of the cell container to the lithium anode in the cell of Fig. 6.

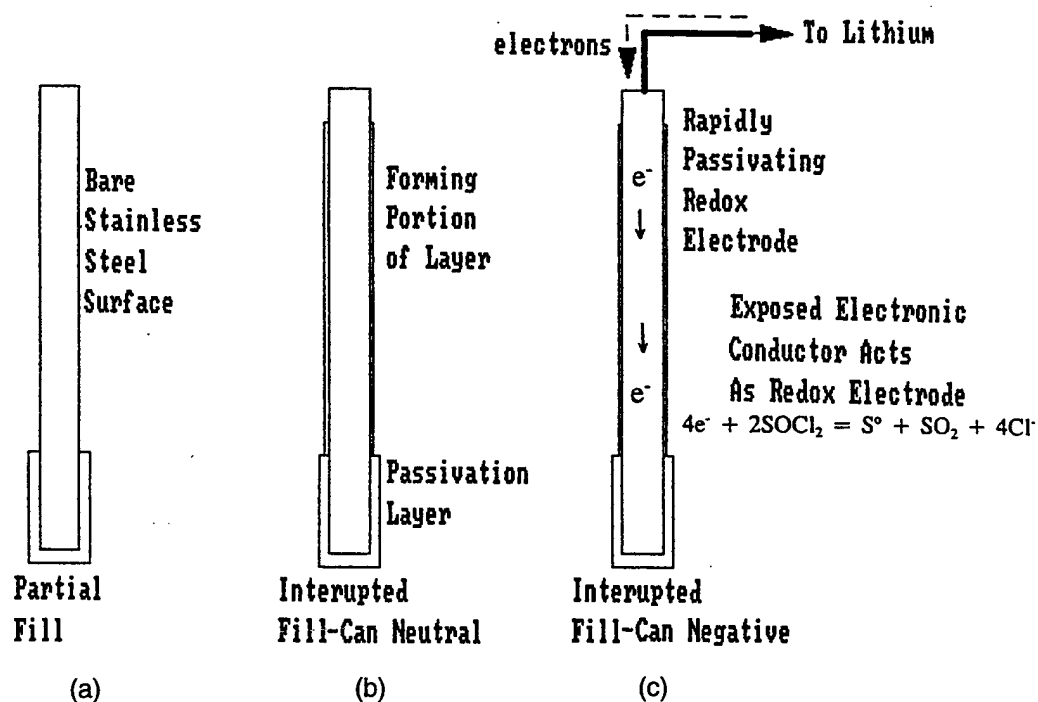


Figure 12. Schematic depicting the chemical and electrochemical reactions taking place on the surface of the stainless steel resulting from an interruption in the addition of electrolyte.

4.3 Belated Touching of the Can

This configuration was investigated because as the cell is filled, it is possible that the incoming electrolyte first contacts the lithium and cathode surfaces. Following this, electrolyte would begin to wet the stainless-steel container. In this situation, as well as during an interrupted filling process, new areas of the stainless and metallic lithium will have newly exposed surfaces of material wetted by thionyl chloride. When this happens, a combination of processes will take place whereby the newly wetted surfaces of both the container material and the lithium will function as rapidly passivating redox cathodes. In both of these situations, the stainless steel functions as a surface on which the thionyl chloride could be reduced (Figure 12c). This would result in a depression of the lithium electrode potential. Figure 13 shows a very pronounced depression of the overall cell voltage as the concurrent transient current between the lithium and the stainless steel is recorded.

4.4 Search for Cathode Transient

The carbon cathode material was suspected as having a transient voltage response. Since carbon blacks are usually produced in reducing atmospheres, they might have oxidizable surface sites that could be oxidized by the thionyl chloride when first exposed to the electrolyte. These tests proved to be the most significant ones since the carbon electrodes tested in these studies displayed the most significant voltage transients. Although these electrodes were made by another cell manufacturer, they were felt to be representative of cathodes as used in lithium thionyl chloride cells. The durations of

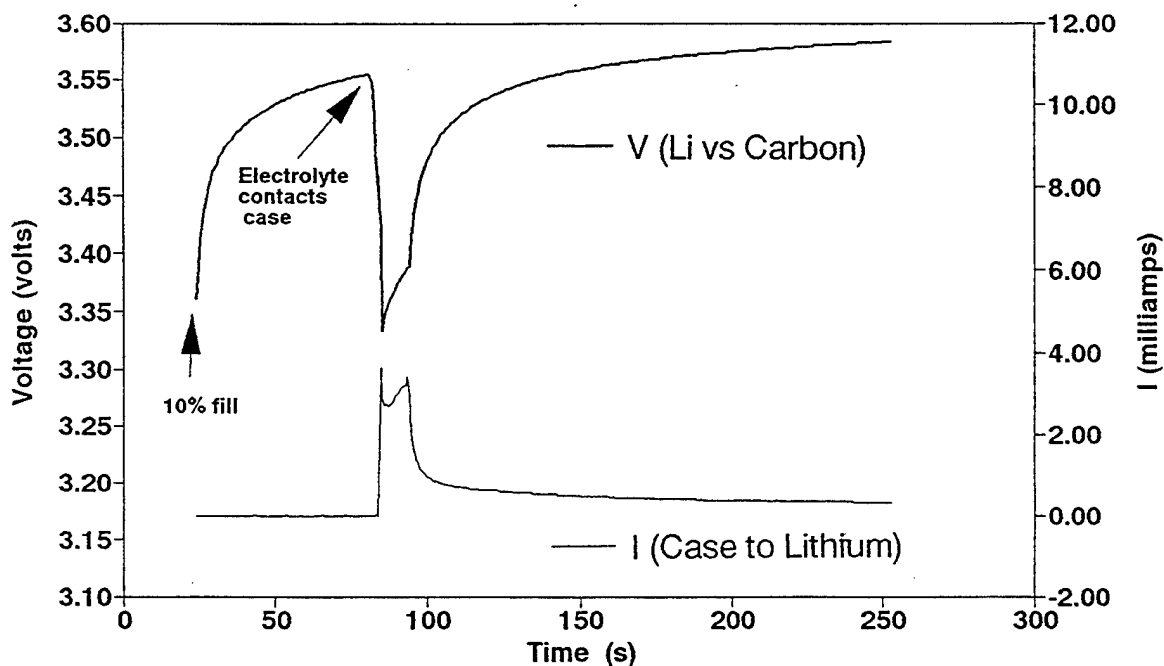


Figure 13. Current and voltage traces following the belated wetting of the cell container by the electrolyte in the cell of Fig. 7.

the voltage transients were much longer than those of the lithium or the stainless steel. Figure 14 shows a typical sequence of filling the experimental cell. It shows a transient voltage dip during the initial 10% fill portion, followed by another dip during the subsequent portion of the filling process. By expanding the voltage scale (Figure 15) and adding the lithium reference electrode potential (Figure 16), it can be seen that the majority of the transient is caused by the cathode. As the reaction products diffuse out into the bulk electrolyte, the voltage will asymptotically approach the standard open-circuit voltage (E°). When new portions of the porous carbon electrode are exposed, and these interior surface sites are oxidized, there will be a rapid, but not instantaneous, drop in the voltage of the cathode portion of the total cell voltage. Following this, there will again be a slow recovery as the reaction products diffuse from within the electrode structure out into the bulk electrolyte. Figure 17 is similar to Figure 14, except the cathode had been used before. There is very little transient response in this electrode. These results suggest that there are oxidizable sites in the surface of the carbon that can be oxidized during the initial exposure to thionyl chloride. Figure 18 depicts the internal oxidation reactions as the thionyl chloride oxidizes the surface sites on the carbon.

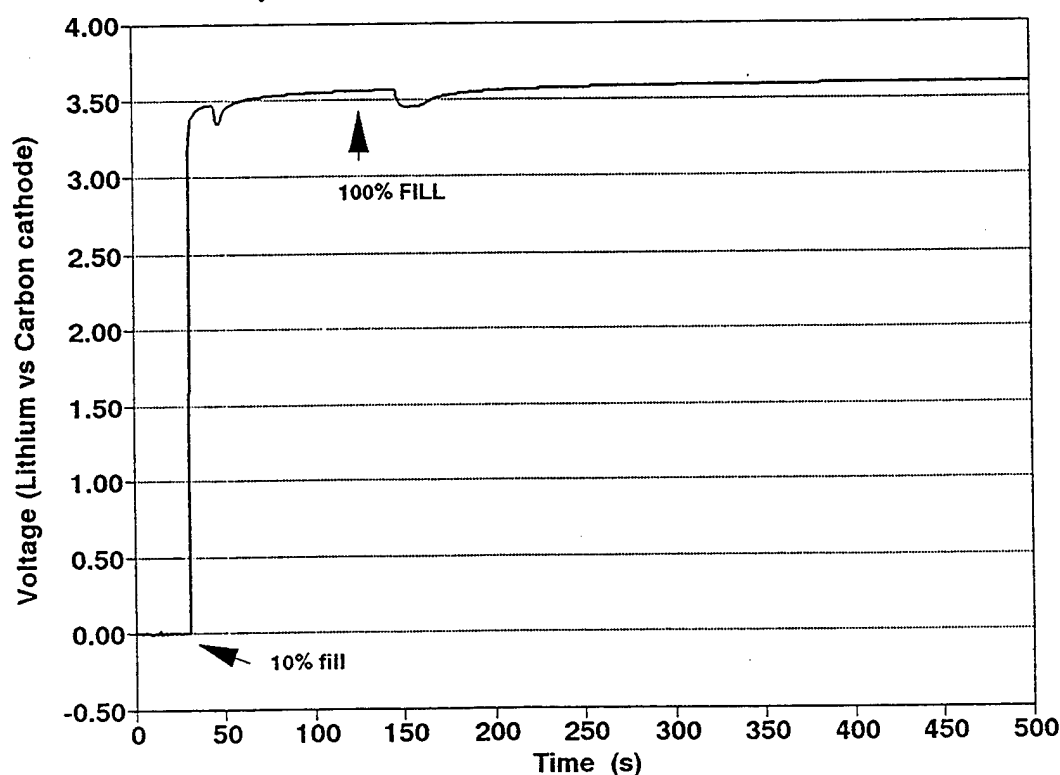


Figure 14. Voltage trace of the cell voltage simulating an interruption of the filling process after about 10% of electrolyte is added to the cell of Fig. 8.

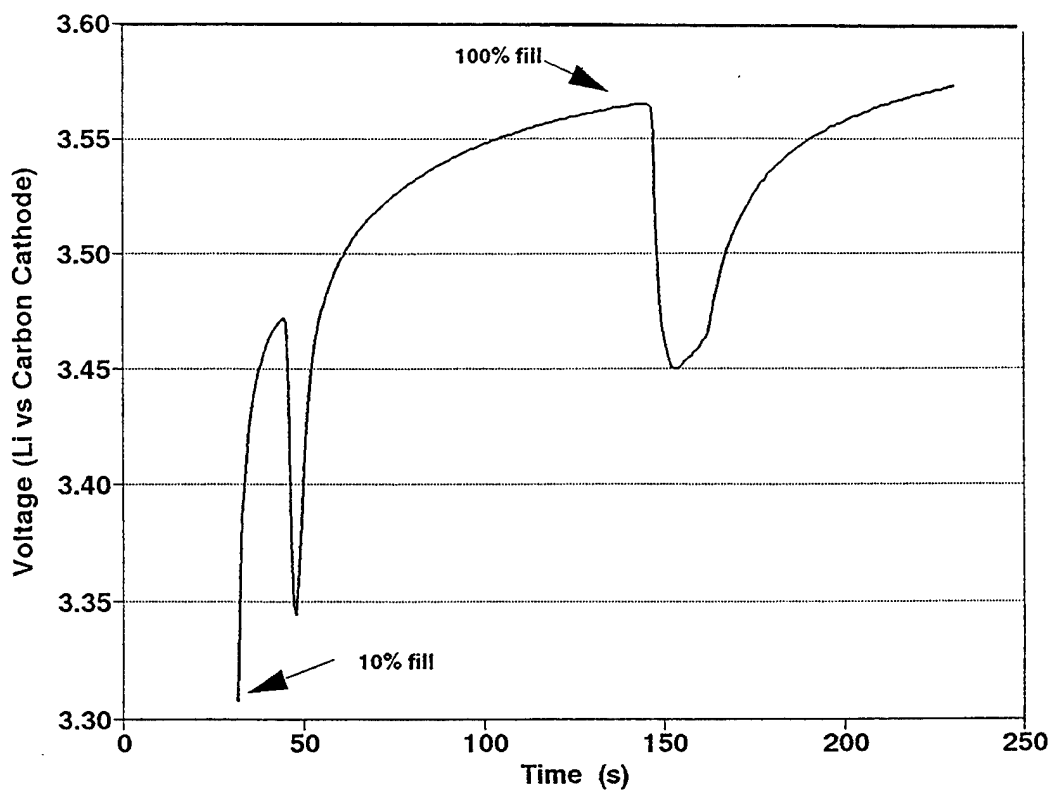


Figure 15. Expanded voltage trace between the reference electrode and the carbon electrode during the first and second stage of the electrolyte addition to the cell of Fig. 8.

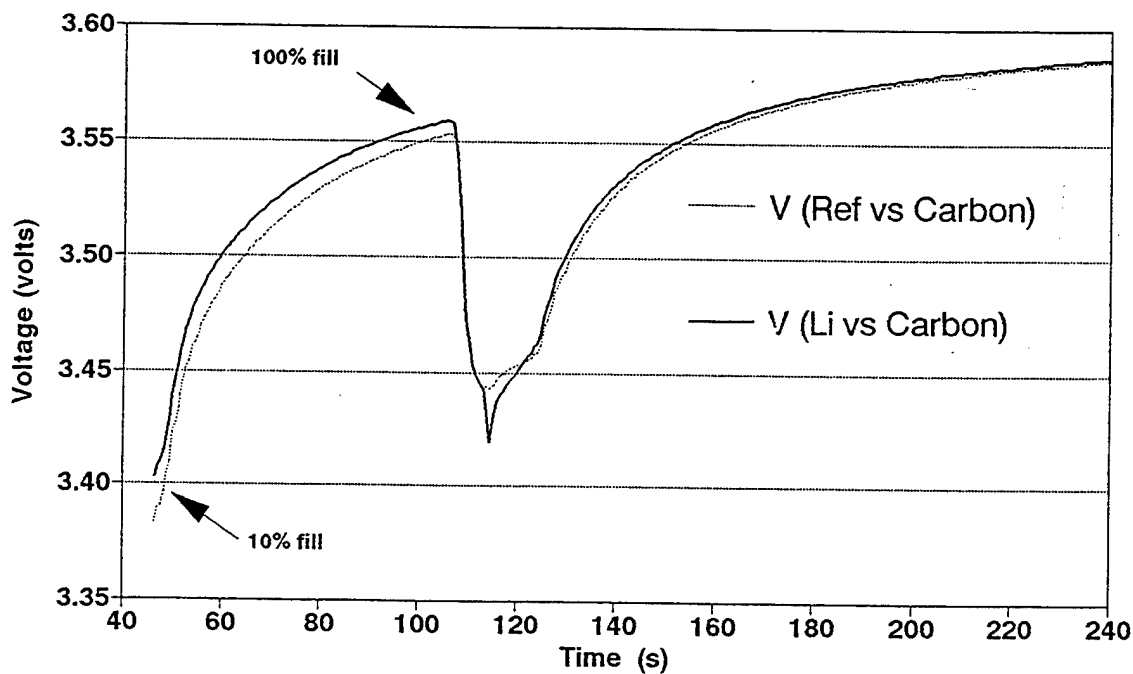


Figure 16. Expanded voltage traces recorded during a repeat of the test examining for cathode voltage transients in the cell of Fig. 8.

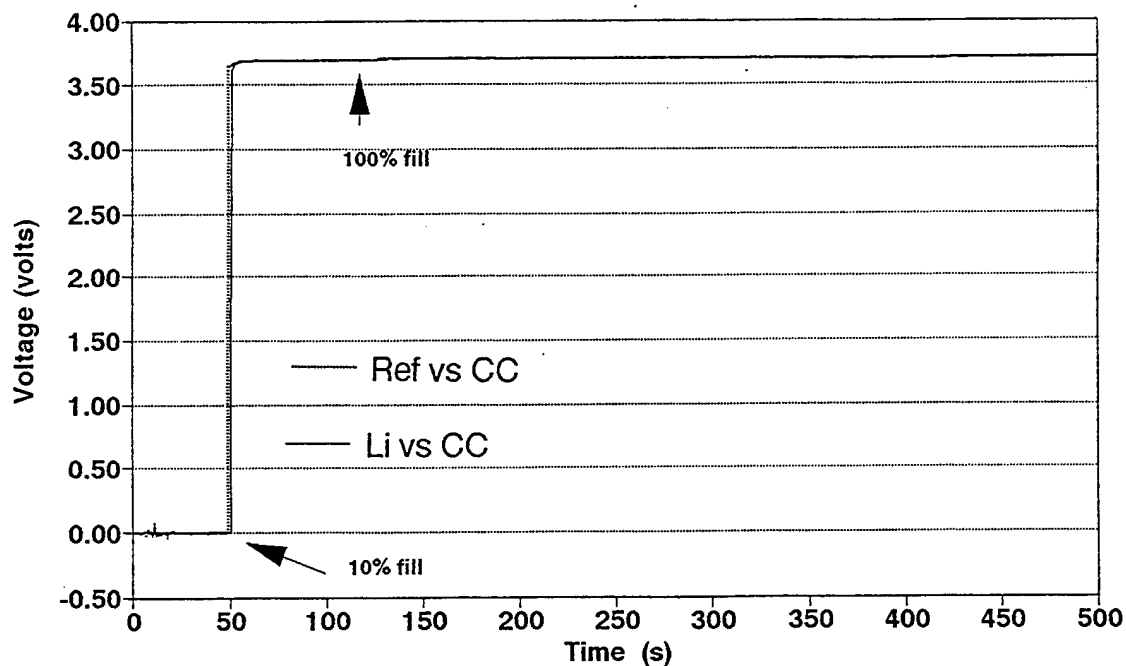


Figure 17. Voltage trace of an interrupted filling process in a cell containing a used cathode.

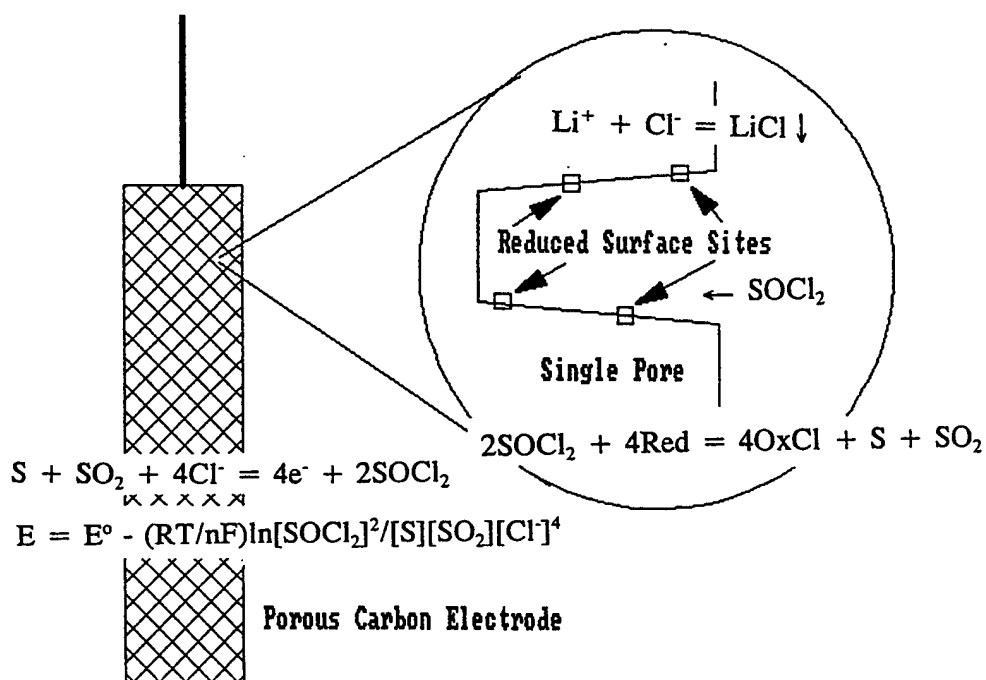


Figure 18. Schematic view of suggested oxidation of interior surface sites of porous carbon electrodes.

5. Explanations of Transients in Full-Sized Cells

With the information gained from the cell tests, the voltage transients seen during both a normal and interrupted filling process can be explained. The two often-seen features of the voltage traces generated during a normal filling process (Figure 1a) are mostly due to the porous carbon cathode material. The short voltage dip occurring a few seconds after the beginning of the inflow of electrolyte to the cell (feature 1) is caused by a reduction of the thionyl chloride redox potential. The electrolyte, after first coming into contact with surface carbon that is relatively free of oxidizable surface sites, enters into the porous structure where some of it is reduced. This temporarily lowers the cathode potential until the products of reaction have time to diffuse out into the bulk electrolyte. At the same time, the portions of the cell container that are wetted by the electrolyte function as redox electrodes and cause the lithium electrode to undergo electrochemical discharge. This also causes a reduction in the overall cell voltage (Figure 1a).

The short voltage dip that coincides with the cessation of electrolyte flow and the return of the internal portions of the cell to atmospheric pressure (feature 2) is also due to the carbon electrode. Lithium thionyl chloride cells are filled by first applying a vacuum to the internal portion of the cell and then allowing the electrolyte to be drawn in under the created pressure differential. When the filling reservoir has been emptied, the ambient pressure within the dry box enters the cell. This pressure step pushes the electrolyte further into the cell where it contacts more of the interior of the carbon electrode. More of the lithium surface may also be exposed to electrolyte as well, but the response time of the lithium appears to be much quicker than that of the carbon electrodes. It only takes a few seconds to reestablish the passivation layer on these portions of the lithium surfaces (Figure 9).

The approximately 5-min out-of-family signature of the large production cell in question (feature 3) can be attributed to voltage transients as already described. This transient followed a 12-min cessation of electrolyte flow. The first downward sloping portion with a slope greater than the following downward sloping portion can be explained as caused by new areas of the stainless steel, lithium, and carbon being exposed to electrolyte. The shallower portion of the trace that follows indicates that the flow rate is lower during this portion of the filling than in the preceding one. The normal time required for the electrolyte to flow into these cells is just over 1 min. The fact that the reinitiated filling lasted about 4 min indicated that although the obstruction within the needle had been removed, the normal flow rate was not fully restored. The short dip as per Figure 1b takes place at the apparent end of the filling process that in total took about 19 min from the time the filling began.

6. Conclusions

A large-capacity lithium chloride primary cell had an interruption in its filling process caused by blockage of the filling needle. It is believed that the blockage consisted of lithium chloride crystals that were in the filling tank as a result of an incomplete washing and flushing at the conclusion of some previous filling procedures. Once the blockage had taken place and following an obligatory waiting period, an operation that restarted the flow of fluid into the cell was carried out. The subsequent flow rate, although lower than normal, did allow all of the electrolyte to enter the cell. Other cells with intentional or unintentional interruptions in the filling procedure were shown to display transient decreases in the cell's open-circuit voltage. Following extensive discussions relative to the internal chemical and electrochemical reactions taking place during the first few minutes after being exposed to the electrolyte, explanations of the out-of-family signature could be made that did not involve the existence of a transient internal short circuit.

TECHNOLOGY OPERATIONS

The Aerospace Corporation functions as an "architect-engineer" for national security programs, specializing in advanced military space systems. The Corporation's Technology Operations supports the effective and timely development and operation of national security systems through scientific research and the application of advanced technology. Vital to the success of the Corporation is the technical staff's wide-ranging expertise and its ability to stay abreast of new technological developments and program support issues associated with rapidly evolving space systems. Contributing capabilities are provided by these individual Technology Centers:

Electronics Technology Center: Microelectronics, VLSI reliability, failure analysis, solid-state device physics, compound semiconductors, radiation effects, infrared and CCD detector devices, Micro-Electro-Mechanical Systems (MEMS), and data storage and display technologies; lasers and electro-optics, solid state laser design, micro-optics, optical communications, and fiber optic sensors; atomic frequency standards, applied laser spectroscopy, laser chemistry, atmospheric propagation and beam control, LIDAR/LADAR remote sensing; solar cell and array testing and evaluation, battery electrochemistry, battery testing and evaluation.

Mechanics and Materials Technology Center: Evaluation and characterization of new materials: metals, alloys, ceramics, polymers and composites; development and analysis of advanced materials processing and deposition techniques; nondestructive evaluation, component failure analysis and reliability; fracture mechanics and stress corrosion; analysis and evaluation of materials at cryogenic and elevated temperatures; launch vehicle fluid mechanics, heat transfer and flight dynamics; aerothermodynamics; chemical and electric propulsion; environmental chemistry; combustion processes; spacecraft structural mechanics, space environment effects on materials, hardening and vulnerability assessment; contamination, thermal and structural control; lubrication and surface phenomena; microengineering technology and microinstrument development.

Space and Environment Technology Center: Magnetospheric, auroral and cosmic ray physics, wave-particle interactions, magnetospheric plasma waves; atmospheric and ionospheric physics, density and composition of the upper atmosphere, remote sensing using atmospheric radiation; solar physics, infrared astronomy, infrared signature analysis; effects of solar activity, magnetic storms and nuclear explosions on the earth's atmosphere, ionosphere and magnetosphere; effects of electromagnetic and particulate radiations on space systems; space instrumentation; propellant chemistry, chemical dynamics, environmental chemistry, trace detection; atmospheric chemical reactions, atmospheric optics, light scattering, state-specific chemical reactions and radiative signatures of missile plumes, and sensor out-of-field-of-view rejection.



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